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LAMELLAR COMPOSITES FORMED BY SPUTTER DEPOSITION

R. A. Busch, et al

Battelle-Pacific Northwest Laboratories

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The initial work on the two concepts of the formation of lamellar composites by sputter deposition is presented.

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Principal Investigator
H. R. Gardner
509-942-2260

Program Managers
R. A. Busch
509-942-2685

J. W. Patten
509-942-2603

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Wm. Walker

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INTRODUCTION

This report describes the progress made during the first half of Phase I of the investigation of lamellar composites formed by sputter deposition of alternate layers of selected two component systems. Phase I is the first of a proposed three-year, three-phase program being conducted by Battelle Northwest Laboratories.

The purpose of this investigation is to evaluate the potential of two approaches to produce the lamellar composities. In each approach the favorable properties of both components will be used to obtain properties which neither component has initially. In one approach a thin-layered system is being explored in which strengthening is derived from the interface effects and small thickness of individual layers. In the other approach a thicker layered compound-forming system is being explored. This system is strengthened by the bulk properties of the component materials and their reaction products as modified by the constraints of bonding to each other. The dividing line in layer thickness between the two systems is felt to be about 1000 Å.

Both types of composites are expected to exhibit yield strengths on the order of one percent or more of the elastic modulus of the composite material. The range of mechanical properties obtainable is expected to depend on the difference in elastic modulus between the component materials, the relative and absolute thickness of the layers, and the deposition conditions employed.

Sputter deposition offers a unique process for producing lamellar composites It can precisely and reproducibly control individual component layer thicknesses down to tens of atom layers. This can provide control over macroscopic properties

through layer thickness effects. A second feature of sputtering is its ability to achieve interlayer bond strengths of the order of the material strength and thereby attain good load transfer across the matrix interface. With respect to production, a single sputter-deposition apparatus can form a deposit of one millimeter thickness over an area of approximately 500 cm^2 in a 24-hour day.

In Phase I of the thin-layered system investigation, lamellar composites of alternate pure copper and molybdenum layers have been sputter deposited. Effects on the mechanical properties of such parameters as absolute and relative layer thickness, deposition temperature and bias, and heat treatment after deposition are now being evaluated.

In the compound-forming system program, criteria have been established for the selection of material pairs which have useful properties in lamellar composite form. The material pair which best satisfied these criteria was formed from beryllium and titanium. Small-scale depositions of this pair are currently being made for evaluation.

SUMMARY

TECHNICAL PROBLEM

The objective of the program is to develop new composites which utilize the favorable properties of the materials they combine to produce properties which neither component originally has. Mechanical properties and their thermal stability are of primary interest for assessment of usefulness in engineering applications.

Thin-Layered Systems

Koehler[1] has proposed the design of a strong solid composed of alternate layers of materials with high and low elastic constants. He expects yield strengths on the order of one percent

of the elastic modulus if layers are thin enough (~200 Å) to prevent the operation of Frank-Reed sources. It also has been shown[2-4] that up to 20 Å of a pure metal may assume the crystal structure of another metal upon which it has been deposited. This is referred to as pseudomorphic or epitaxial growth. The boundary region of such a film would be very highly stressed in the same manner as are Guinier-Preston zones or boundaries of other coherent precipitates.

The object of the thin-layered system program is to fabricate lamellar composites with layers less than 200 Å thick. These layers would consist of two metals mutually insoluble in the solid state, with different crystal structures and with widely different elastic moduli and coefficients of thermal expanison. Contributions to a high level of internal stress should be realized from lattice distortion produced by pseudomorphic layers, fine grain size and high internal stress levels achievable in pure sputtered materials, and property mismatch. It is not expected that Frank-Reed sources will operate within a given layer; therefore, dislocations should be very difficult to generate. The long-range nature of the dislocation barriers (layer interfaces) together with the need to preserve Burger's vector in moving across an interface should also make dislocation movement very difficult. Vacancy diffusion to the interfaces and subsequent defect movement should provide the only deformation mechanism.

Compound-Forming Systems

Compound-forming systems do not depend on the small dimensions of the layers for their properties. The elements or compounds forming the layers are selected for their individual properties and their compatibility with each other. The reinforcing layer consists of a high elastic modulus material such as an oxide,

carbide or intermetallic compound; the other layer is a metal with a reasonable degree of plasticity. These composites could function similarly to existing composite materials in that the metal would protect, orient, and transfer load to the reinforcing layer. They would be expected to achieve higher strengths than existing composites due to the strong bonding between layers. They may, however, suffer the drawback of a potential continuous crack path in the brittle reinforcing layer.

GENERAL METHODOLOGY

The general approach is experimental. Tensile testing and microhardness techniques are being used to study mechanical properties as a function of relative and absolute laminate layer thickness, substrate temperature and bias during deposition, and heat treatment temperature after deposition. In addition such techniques as x-ray diffraction, scanning electron microscopy, calorimetry, and resistivity measurement are being used to gain insight into structure, fracture behavior, and other composite characteristics as a function of time and temperature.

Depositions on large cylindrical substrates will be performed to provide tensile samples and additional material for metallurgical characterization.

TECHNICAL RESULTS

Thin-Layered Systems

Nine sputter-deposition experiments have been conducted from which six copper-molybdenum lamellar composites were obtained. The composites were in sheet form approximately 62 cm long by 12 cm wide and up to 0.076 cm thick. Temperatures from 65 to 450°C and layer thickness ratios from 1/1 to 3.9/1 (Cu/Mo) were represented. Samples were heat treated from 1 to 4 hours at

500, 750 and 1000°C. Evaluation of these deposits has begun and preliminary data indicate that very high hardness and brittle fractures are to be expected in these materials. Transmission electron microscopy indicates that a very high integrity layered structure has been obtained. This preliminary data implies that thin laminate structure does provide a new strengthening mechanism.

Compound-Forming Systems

Criteria were established for the selection of material pairs with useful properties in lamellar composite form. Eight pairs were selected. Seven were based on beryllium combined with chromium, magnesium, molybdenum, tantalum, titanium, tungsten, and zirconium; and one was composed of magnesium and silicon. Although no system satisfied all the criteria, the Be-Ti composite best satisfied the criteria. It was, therefore, selected for the initial work.

A target arrangement capable of forming the desired intermetallic composition in one layer of a pair with either pure Be or Ti in the other layer was designed and fabricated. Parameters for these depositions are being determined by small-scale depositions in another apparatus to ensure, if possible, that the large deposits will not be excessively brittle or warped by internal strains and will therefore be adequate for testing.

THIN-LAYERED SYSTEMS

MATERIALS AND PROCEDURES

Deposition

Sputtering targets were fabricated from 99.99% pure electrolytic tough pitch (E.T.P.) copper and 99.95% pure molybdenum. Substrates were rolled OFHC copper, 61.8 cm x 12 cm. Substrates used for initial deposits were 0.051 cm thick and had a surface

finish of approximately 0.8 microns. From the rolling operation, they had small grooves oriented parallel to the long dimension of the substrate. Substrates used for later deposits were 0.064 cm thick and had a surface finish of better than 0.2 microns. The properties of the Cu and Mo used as components to form the lamellar composites are listed in Table I.

TABLE I. Properties of the Components of the Lamellar Composite

Property	Cu	Mo
Density (g/cm^3)	8.96	10.2
Melting Point (^0C)	1083	2625
Thermal Expansion ($/^0C$)	16.5×10^{-6}	4.9×10^{-6}
Latent Heat of Fusion (cal/g)	50.6	70
Electrical Resistivity (μ ohm-cm)	1.6730	5.17
Modulus of Elasticity (kg/cm^2)	1.2×10^6	3.5×10^6
Crystal Structure	fcc	bcc
Lattice Constant = a(kX)	3.6080	3.1400

A diagram of the sputtering apparatus is illustrated in Figure 1. In operation the substrate rotates around the half-cylinder Cu and Mo targets so that a given area on the substrate is exposed to the Cu target for the first half of a revolution and to the Mo target for the second half of the revolution. Deposit layer thickness is determined by substrate-rotation and sputter-deposition rates. In experiments to date the rotation rate has been fixed at 44 rpm. The deposit (substrate) temperature is controlled by contact with a water-cooled substrate holder. Temperature is measured with a thermocouple which penetrates the substrate holder and contacts the Cu substrate.

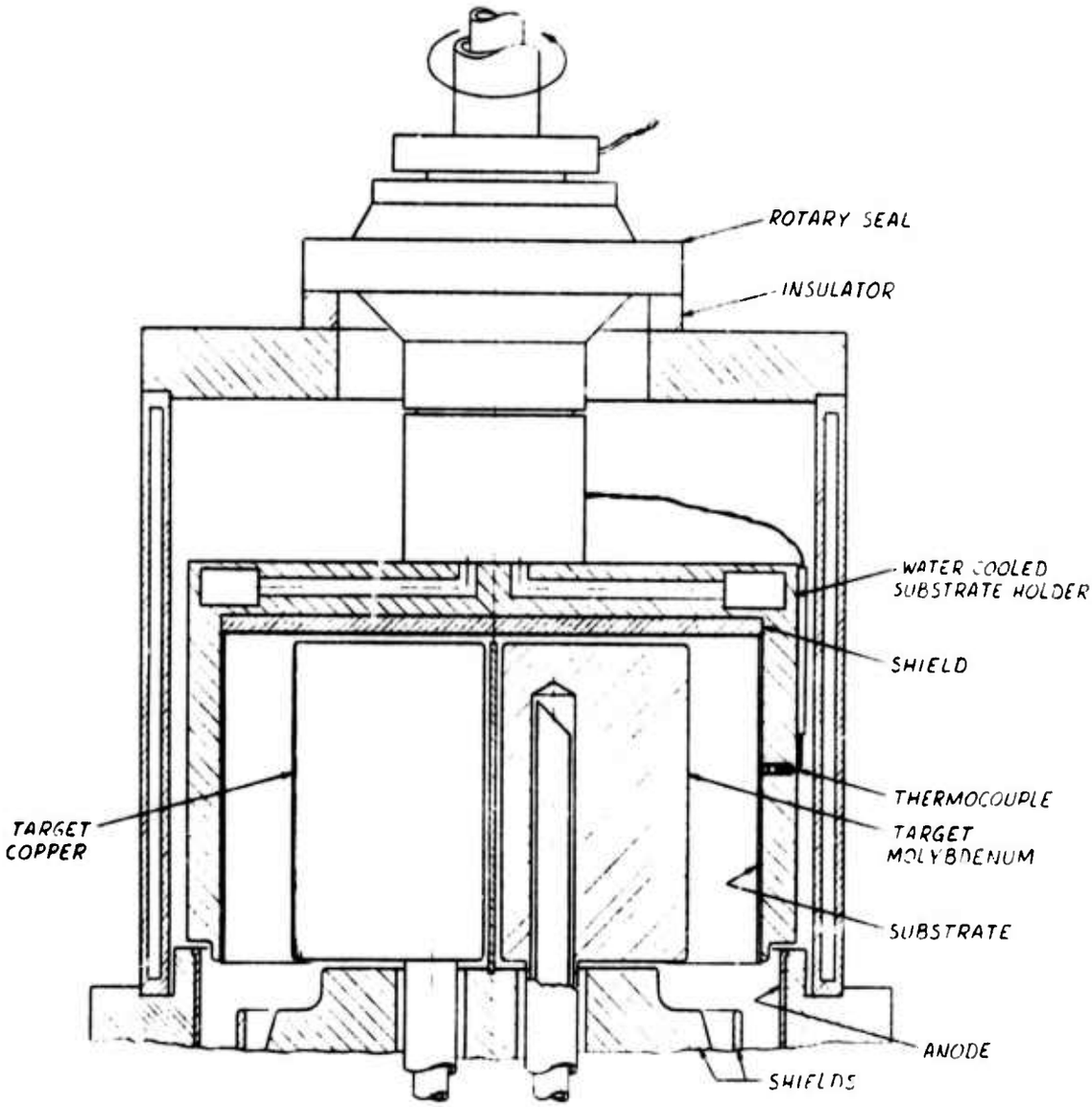

FIGURE 1. Sputtering Hardware for Deposition of Cu-Mo Lamellar Composites

The sputtering system is typically evacuated with a liquid nitrogen trapped oil diffusion pump to 9×10^{-7} torr for 18 hr before deposition begins. Then high-purity krypton sputtering gas is admitted to the sputtering chamber until a pressure of 3 μ is achieved. The Cu substrate is etched by ion bombardment with 100 v Kr^+ ions for approximately 10 min to produce an atomically clean surface. During sputter deposition the voltages applied to the Cu and Mo targets are adjusted independently to obtain the desired sputtering rates. It was found that substrate bias voltage could not be varied effectively in this apparatus. Substrate potential, therefore, was very close to 35 volts (plasma related or floating potential) for all deposits.

The history of deposits made to date is summarized in Table II.

Heat Treatment

Specimens were heat treated for various times at temperatures up to 1000°C in a vacuum furnace capable of 1×10^{-6} torr at 1000°C. A summary of heat-treatment conditions is given in Table III. Deposit TLC-2 was chosen for the treatment because of its high quality and deposit TLC-4 was chosen because it was thick enough for transmission electron microscopy.

Evaluation

Nickel-filtered Cu K_α radiation in an x-ray diffractometer is being used to determine structure of as-sputtered and heat-treated specimens. Fifty percent of the x-ray intensity is produced by the first 15,400 Å of the surface. This provides ample x-ray penetration to examine many layers and pseudomorphic regions.

A thinning technique has been developed for Cu-Mo laminates that allows the use of a 200-kV electron microscope to examine layer structure. This technique is being used to examine deposits in cross section but it requires at least a 0.051-cm-thick deposit.

TABLE II. Deposition History and Comment

Deposit Number	Substrate Surface	Deposit Thickness (cm)	Temperature (°C)	Approximate Layer Thickness (Å) Cu	Mo	Substrate Etched	Deposition Interruption	Deposit Surfaces	Shields	Comments
OTLC-1	Rough	None	---	---	---	No	---	---	S.S* & Cu	Faulty assembly
OTLC-2	Rough	0.076	65	50	50	No	Many	Good, some defects	S.S. & Cu; peeling	Electrical problems limited length of run.
OTLC-3	Rough	None	---	---	---	No	---	---	---	Electrical problems terminated run at its startup.
TLC-1	Rough	None	65	---	---	No	---	---	S.S. & Cu; peeling	Shield peeling produced shorts and terminated run.
TLC-2	Rough	0.025	65	50	50	Yes	One after 2 hours	Good, very few defects	S.S. & Cu; peeling	Shield peeling produced shorts and terminated run.
TLC-3	Rough	0.028	450	50	50	Yes	None	Good, few defects	S.S. & Cu; peeling	System vented to air at 450°C; deposit badly oxidized; shield peeling produced shorts and terminated run.
TLC-4	Rough	0.025	95	50	50	Yes	None	Good, few defects	S.S., Cu and Ta; bad peeling	Shield peeling produced shorts and terminated run.
TLC-5	Smooth	0.036	400	93	24	Yes	None	Good, few defects	S.S. & Cu; peeling	Shield peeling produced shorts and terminated run.
TCL-6	Smooth	0.069	400	50	50	Yes	None	Good, few defects	Cu; peeling	Shield peeling produced shorts and terminated run.

*S.S. = 304 stainless steel

TABLE III. Heat-Treatment Parameters for Deposits TLC-4 and OTLC-2

Temperature (°C)	Time (hrs) 1	2	3	4
500	TLC-4	TLC-4	OTLC-2	TLC-4
750	TLC-4	OTLC-2 TLC-4	OTLC-2	TLC-4
1000	TLC-4	OTLC-2 TLC-4		OTLC-2 TLC-4

ASTM E8 subsize specimens are being fabricated from the deposits with a Tensilegrind machine. The Cu substrate is machined from the deposit in the 0.625 cm × 25.0 cm gauge length which is then polished. Strain gauges are cemented to both sides of its length. Tensile tests are performed at room temperature in an Instron Model TTC tensile testing machine.

Fracture surfaces are being examined in a scanning electron microscope to determine mode of fracture and its relation to composite structure.

Microhardness of deposits is being measured at room temperature in both as-deposited and heat-treated conditions. In addition, hot hardness is being measured by Professor John Moteff at the University of Cincinnati as a function of time at temperatures to 1000°C. An inert-atmosphere cantilever-beam-loading apparatus is used for this work. Hardness results will be used to estimate tensile properties for a wide range of conditions.

Auger electron spectroscopic analysis is being performed by Professor Gotfried Wehner at the University of Minnesota on as-deposited specimens and specimens heat treated for various times at temperatures to 1000°C. Results of this investigation should indicate extent of interdiffusion between Cu and Mo layers.

Stored energy release investigations are being carried out in a Perkin-Elmer DSC-2 scanning calorimeter with a maximum temperature capability of 725°C. Information obtained from these investigations should allow prediction of annealing mechanisms and activation energies and, therefore, prediction of mechanical behavior for extended times at elevated temperatures.

Resistivity measurements are being made in a Battelle-Northwest designed and constructed apparatus using a four-probe technique and a digital data acquisition and analysis system. Objectives are the same as for the calorimetric investigation described above.

RESULTS AND DISCUSSION

Deposition

Shield peeling has been a problem in all depositions. Various precautions were taken to enhance adherence between sputtered material and shields. These included cleaning the shields in various solvents and bead blasting, machining, and vacuum baking them before deposition. Different shielding materials (stainless steel, tantalum, and OFHC copper) were tried and evaluated as a possible solution. OFHC copper was found to be the most satisfactory. Peeling was successfully eliminated in all areas except on shields near the filament.

Deposits made at 65 and 95°C were very highly stressed. When they were removed from the cylindrical substrate holder, these substrates straightened out in the 61.8-cm dimension (the cylinder circumference) and warped approximately 1 cm in the 12-cm dimension (parallel to the cylinder axis) towards the

deposit. This behavior is illustrated in Figure 7. Deposits made at 400 and 450°C retained the shape of the cylindrical substrate holder.

Evaluation

Evaluation of these deposits is in the early stages; all of the necessary equipment is available. Satisfactory procedures have been established in most instances, but only preliminary data, as presented below, are available at this time.

Low temperature deposits examined to date (TLC-2 and TLC-4) were heavily textured with (111) Cu and (110) Mo planes oriented parallel to the deposit-substrate interface. The only high temperature deposit which has been examined (TLC-3) was heavily textured in the Mo layers with (110) Mo planes oriented parallel to the deposit-substrate interface. The Cu layers were slightly textured with (100) Cu planes oriented parallel to the deposit-substrate interface. This corresponds to a recrystallization texture previously observed in sputtered Cu.[5] No evidence of structure other than fcc Cu and bcc Mo has been observed.

Figure 3 shows photographs taken by a transmission electron microscope of cross sections from deposit OTLC-2. Only Mo layers are visible and these were too thick for electron transmission. The Cu layers were removed during thinning, as evidenced in Figure 4, which is an electron diffraction pattern of a region similar to those shown in Figure 3. Note that only bcc Mo rings are present (d spacing may be calculated from d=5.354/diameter of ring in cm). These photographs provide direct evidence for continuous layers of Cu and Mo. Each of these layers was about 50 Å thick, the layer thickness sought when the sputtering system was designed and the sputtering parameters selected. Layers of this thickness are ideal for the purposes of this program and are being used as the standard for successive runs.

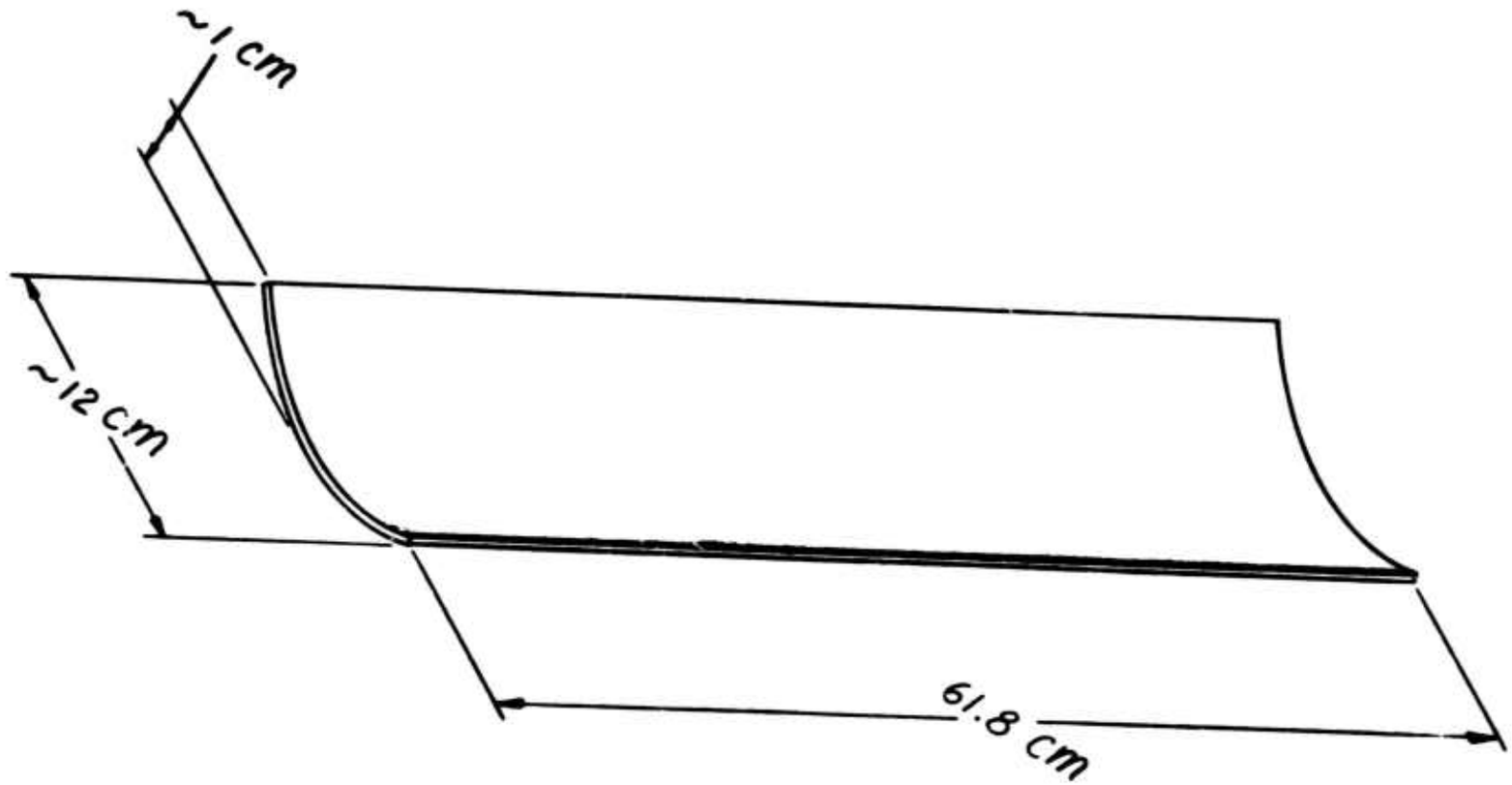

FIGURE 2. Approximate Configuration of 65 and 95°C Deposits After Removal from the Substrate Holder

Preliminary results on TLC-5, which is the only deposit tested to date, indicated no plastic deformation. The elastic modulus was 1.2×10^6 kg/cm^2, which is typical of pure Cu. Specimen TLC-5 was deposited at 400°C with thicker Cu layers than Mo layers (see Table II); brittle failures in this deposit indicate that they can be expected in deposits not as yet tested. The modulus for other deposits, however, should be higher. Fracture appears to occur at columnar boundaries originating from growth behavior. These are not necessarily grain boundaries.[6]

Hardness of TLC-4 as a function of heat-treatment time for various heat-treatment temperatures is shown in Figure 5. Measurements of the room temperature hardnesses of other deposits are not yet available. Hot hardness is being measured as a function of time at four temperatures between 500 and 1000°C.

Evaluation of specimens utilizing Auger, calorimetry and resistivity methods are in progress.

Layer Thickness ∿ 58Å. 198,000X

Layer Thickness ∿ 47Å. 450,000X

FIGURE 3. Transmission Electron Micrographs of Cu-Mo Deposit OTLC-2 Illustrating Layer Thickness 200kV Electron Beam

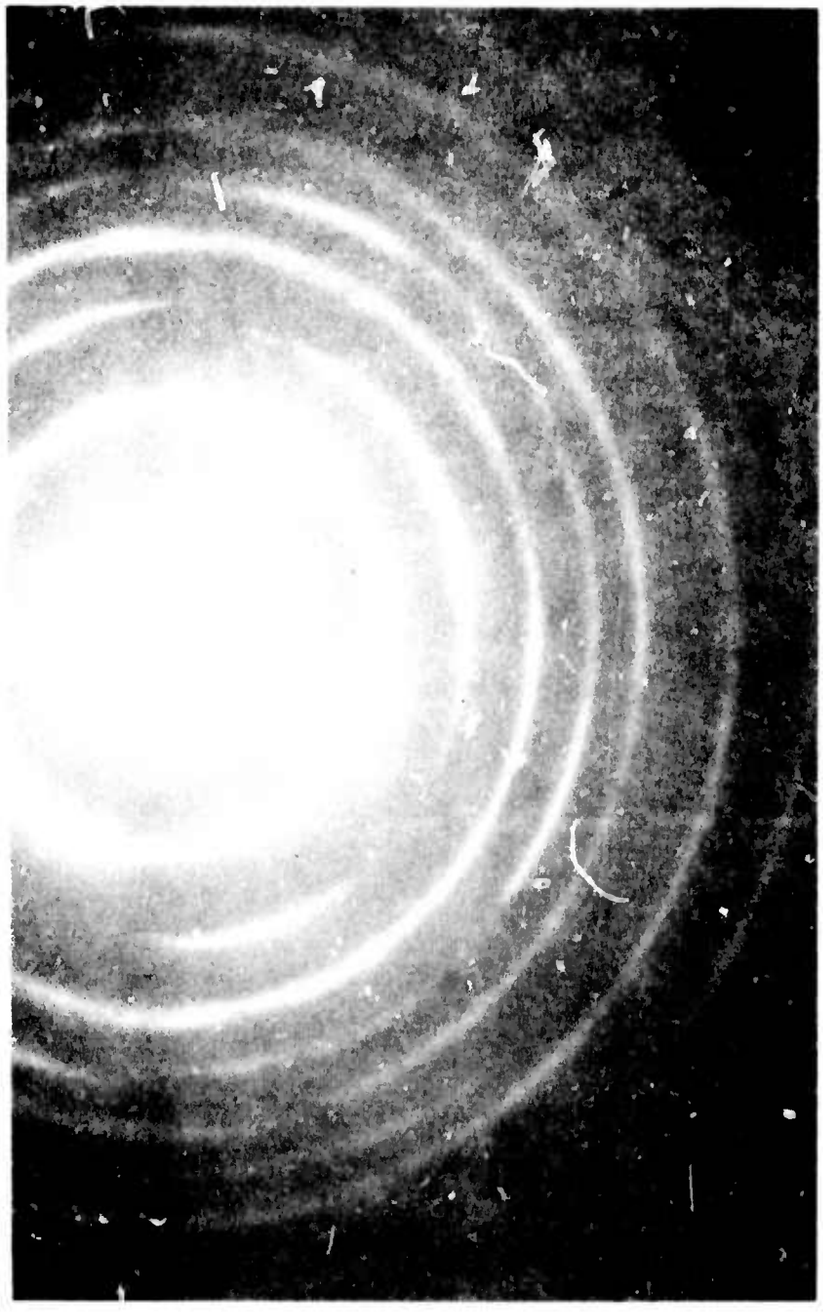

FIGURE 4. Electron Diffraction Pattern of Deposit OTLC-2 Illustrated in Figure 3. Only the Mo Lines Are Visible

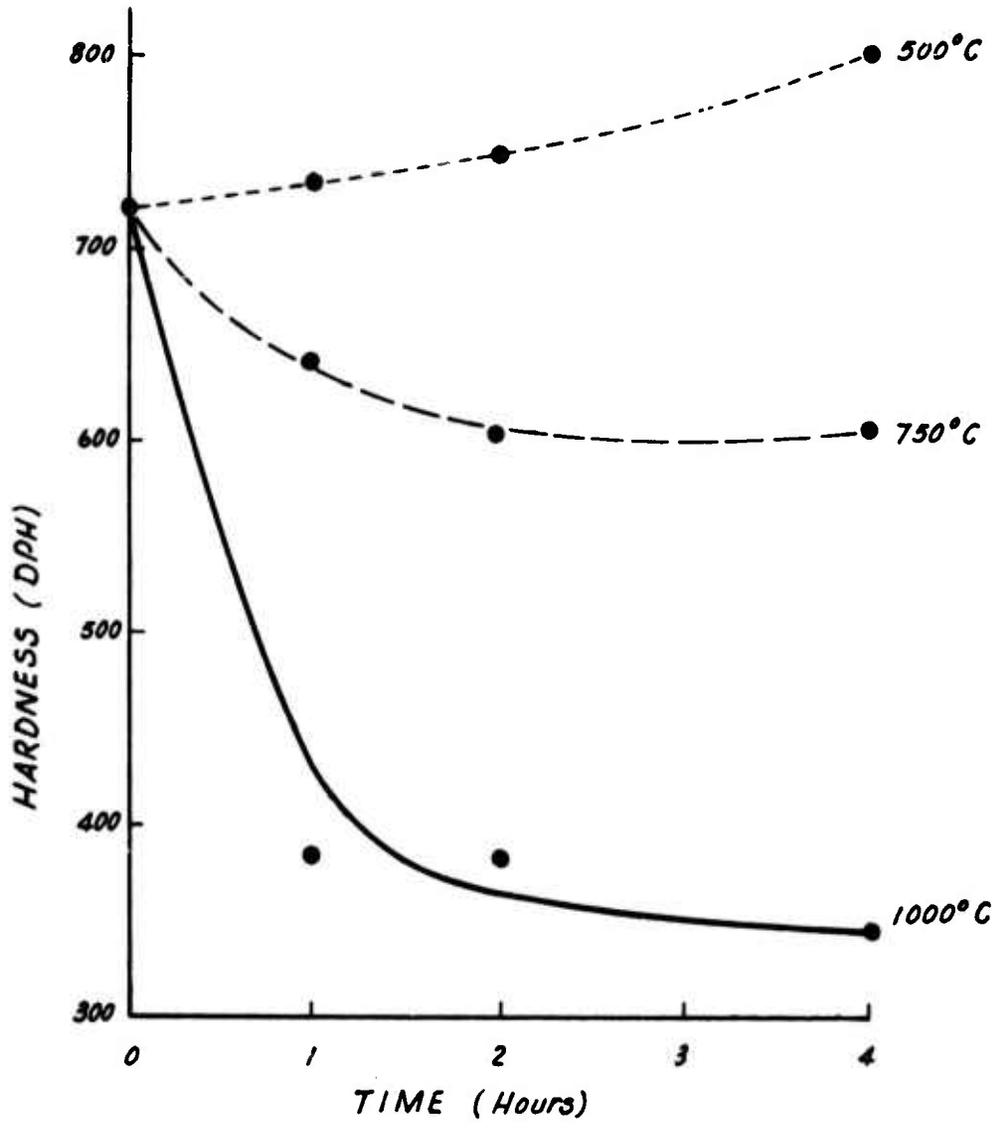

FIGURE 5. Hardness as a Function of Heat-Treatment Time for Various Heat-Treatment Temperatures for Deposit TLC-4

COMPOUND-FORMING SYSTEMS

SELECTION AND EVALUATION OF MATERIAL PAIRS

To determine the tensile properties of lamellar composites made up of metallic and intermetallic layers, criteria were first established for the selection of material pairs which could reasonably be expected to offer useful properties in lamellar composite form. These criteria were:

1. Maximum mismatch of elastic moduli between the compound and one of its constituents at a high average modulus, i.e., maximum values of $E_{AB}-E_A$ and $\frac{E_{AB}+E_A}{2}$.
2. Minimum density, primarily of the metal to be used as the metallic layer.
3. Closest available matching of thermal expansivity.
4. Formation of a compound with a narrow homogeneity range and a high melting or dissociation temperature.
5. Preferably, this compound should be the only one in the alloy system.

Criterion 1 relates to the potential strength of the composite and with criterion 2 determines the stiffness-to-density and potential strength-to-density ratios. Criteria 3 and 4 relate to the mechanical and thermal stability of the composite, respectively, while 5 is directed toward ease of formation of the composite from an initial structure consisting of the two metals.

Eight material pairs were selected, seven of which involved beryllium. These were Be with Cr, Mg, Mo, Ta, Ti, W and Zr; the remaining system was Mg-Si. Of these, Be-Ti best satisfied the criteria. No system satisfied all the criteria.

RESULTS AND DISCUSSION

Specimens of these materials were assembled into two stacks suitable for long-term annealing at temperatures of 600 and 900°C, respectively, and annealed for ~100 hours. The couples were examined for intermetallic formation by microhardness and electron microprobe traverses. The objective of this work was to determine the final product of the intermetallic reactions, i.e., if one or more compounds would be formed, their relative thicknesses, and the existence of interlayer cracking or porosity (Kirkendall effect). The results of the first set of diffusion anneals were not satisfactory, apparently due to excessive oxidation. A second set of anneals was performed with somewhat similar results, although a limited amount of data was obtained.

At that time it was decided that continued attempts to select a material pair by diffusion experiments were not justified and the Be-Ti system was selected. A target arrangement was designed which was capable of forming the desired intermetallic composition in one layer of a pair with either pure metal in the other set. The intent was to circumvent potential difficulties in forming the desired compound by diffusion after deposition. The target materials were purchased; the machined targets are expected to be available by November 15.

Four depositions using side by side "D"-shaped elemental targets have been made in small-scale sputtering equipment. The application of voltage to these targets was programmed to form Ti and Be-Ti layers with the composition of the latter corresponding to $Be_{12}Ti$.

The first deposition, which was made on a cold (16°C) substrate, was prematurely terminated when the deposit flaked off the substrate and caused an electrical short circuit. This deposit yielded an x-ray diffraction pattern consisting of strong titanium lines and a few weak lines tentatively indexed as $Be_{12}Ti$. Subsequent experiments have utilized substrates heated to 300°C. No further flaking has been observed. The sputtering data indicates that Be and Ti can be co-deposited without interference from the poisoning phenomenon observed in some other alloy systems.[7,8]

Two of the depositions were made with absolute layer thicknesses of 1.5-μm and 0.25-μm pairs, respectively, at a constant thickness ratio of intermetallic/metal of 0.4. A fourth depositions was made at the 0.25-μm pair thickness, but with a thickness ratio of 0.1. The hardness, x-ray diffraction data, and apparent ductility in flexure of these deposits will be used to determine the deposition parameters for the first large-scale run, which is now scheduled for the third week in November. These parameters will be selected to maximize the "testability" of the deposit, i.e., conditions expected to lead to low stored energy, small intermetallic/metal ratio, and large absolute layer thicknesses.

The small-scale deposits are also being used to evaluate layer stability during thermal exposure by differential calorimetry, x-ray diffraction and metallography. At the time of this writing, the data obtained are not sufficient to warrant discussion.

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